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MECHANICAL PROPERTIES OF BETA-SILICON NITRIDE WHISKER/SILICON NITRIDE MATRIX COMPOSITES*

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Abstract

The mechanical properties of β-Si_3N_4 whisker / Si_3N_4 matrix composites were investigated. Both as received and modified β-Si_3N_4 whiskers were used to reinforce polycrystalline Si_3N_4. Whisker modification consisted of low pressure chemical vapor deposition of BN onto whisker surfaces. The whisker surfaces were characterized by XPS, SEM, and TEM. Composites were fabricated via hot pressing. Mechanical properties of the composites, primarily fracture stress and fracture toughness, were measured and compared to those of unreinforced polycrystalline Si_3N_4. Composite microstructures and fracture surfaces were characterized by SEM.

Introduction

Whisker reinforced ceramic matrix composites have received a great deal of attention for applications as high temperature structural materials in, for example, advanced heat engines and high temperature energy conversion systems. For these applications, where mechanical reliability is critical, the improvements that can be realized in fracture stress and fracture toughness by the addition of whiskers to polycrystalline ceramics are of great interest. The primary role of the whisker reinforcement is to prevent catastrophic brittle failure by activating mechanisms that dissipate energy during crack propagation. Relevant toughening mechanisms include whisker bridging [1-3], whisker pullout [4-6], crack deflection [7-8], and microcrack formation [9-10]. Of particular importance for optimizing the mechanical reliability of these composites is the effect of the whisker / matrix interfacial characteristics on these toughening mechanisms. Thus, the degree of energy dissipation depends on the nature of the interface, which can be controlled largely by the matrix chemistry, the whisker surface chemistry, and the processing conditions.

A potential candidate for high temperature applications is silicon nitride (Si_3N_4). Polycrystalline Si_3N_4 exhibits excellent high temperature mechanical integrity and environmental stability. Despite these attributes, the brittle nature of Si_3N_4 limits its use where mechanical reliability is critical. However, numerous investigators have recently

demonstrated that the mechanical performance of this material can be significantly improved by reinforcing it with single crystal silicon carbide (SiC) whiskers [11-17]. Table I summarizes typical values of the fracture stress and fracture toughness reported in these systems. As shown in Table I, fracture stress and fracture toughness improvements are not consistently realized upon SiC whisker addition. The variability in the expected property improvements is due partly to the nature of the whisker / matrix interface.

Recently, β-Si_3N_4 whiskers have become commercially available on a limited basis, thus providing another potential reinforcement for polycrystalline Si_3N_4. In this article, the results of the initial investigation of the processing and characterization of β-Si_3N_4 whisker / Si_3N_4 matrix composites are reported.

Experimental

(1) Whisker Surface Modification

The β-Si_3N_4 whiskers and α-Si_3N_4 powder utilized in this investigation were UBE-SNWB* and UBE-SN*, respectively. The physical and chemical properties of these materials are shown in Table II [18].

The surfaces of the β-Si_3N_4 whiskers were treated by chemical vapor deposition of boron nitride (BN). Borane ammonia (BH_3NH_3)**, which decomposes upon heating to yield BH_3 and NH_3 gases, was utilized as the precursor for BN. The whiskers and atmosphere powder were placed in separate crucibles in a horizontal tube furnace. The optimum treatment temperature was estimated using a thermodynamic simulation [19] of Si_3N_4 in the presence of BH_3. The condensed phases predicted by the thermodynamic simulation are shown in Figure 1. The treatment temperature was selected at 800°C, since BN begins to decompose above approximately 1100°K. The furnace tube was evacuated and valved off before the temperature was ramped to 800°C in one hour. When the interior temperature reached the decomposition temperature of BH_3NH_3, the internal pressure increased rapidly. To prevent excessive pressure buildup inside the tube, the excess gas was allowed to escape to the atmosphere. The temperature was then held at 800°C for 2 additional hours.

(2) Composite Processing

The powder / whisker mixtures were processed via a slurry mixing technique. The β-Si_3N_4 whiskers (30 volume percent) and α-Si_3N_4 powder were combined with ethanol. One percent sintering aid, magnesium oxide (MgO), was also added as magnesium hexahydrate ($Mg(OH)_2 6H_2O$)†. The mixture was then blended for five minutes in a high speed blender in order to obtain an intimate distribution of powders and whiskers. Powder agglomerates and whisker bundles were broken up by an additional five minute ultrasonic treatment. Finally, the resulting mixtures were dried and sized in preparation for hot pressing.

Composite densification was achieved via hot pressing††. Hot pressing conditions were estimated using a thermodynamic simulation [19]. Two simulations were performed. The first simulation, shown in Figure 2, was for Si_3N_4 in the presence of MgO. Note that above 1800°K liquid forsterite ($MgSiO_3$) is present and that Si_3N_4 begins to decompose above 2100°K. This defines the processing window for hot pressing. The second simulation, shown in Figure 3, was to ascertain the compatibility of Si_3N_4 in the presence of BN. As shown, BN is stable to above 2100°K. Based on these thermodynamic simulations, the hot pressing conditions utilized are shown in Table III. The unreinforced specimens were hot pressed at 1750°C, while the composite specimens were hot pressed at either 1800

or 1825°C.

(3) Composite Characterization

Transmission electron microscopy (TEM)# was utilized to determine whisker microstructure. Whiskers were ultrasonically dispersed in ethanol. TEM specimens were then prepared by affixing a carbon film to a copper grid, followed by deposition of the whiskers from the suspension.

Whisker surface chemistry was determined by X-ray photoelectron spectroscopy (XPS)##. Survey scans were performed from 1000-0 eV using Mg-kα radiation. Following the survey scans, detailed spectra were taken for the Si2p (110-90 eV), C1s (295-275 eV), N1s (408-388 eV), O1s (540-526 eV), and B1s (202-188 eV) peaks.

Phase identification was performed by X-ray diffraction (XRD)[$]. Powder samples were analyzed using Cu-kα radiation from 10° to 80° 2θ.

Scanning electron microscopy (SEM)[$$] was utilized to examine whisker morphology and to characterize operative toughening mechanisms. Whiskers morphology was examined by mounting whiskers onto an aluminum stub with a graphite suspension and coating with carbon. Toughening mechanisms were determined by examining fracture surfaces of specimens utilized to determine the fracture stress. Specimens for fractography studies were mounted on aluminum stubs and coated with carbon.

(4) Mechanical Property Evaluation

Fracture stress was determined in 4-point bending on 3.2 X 3.2 X 25.4 mm bars at a crosshead speed of 0.2 mm/minute. The outer span was 19.1 mm and the inner span was 6.4 mm. Tensile surfaces were finished with 15 μm diamond paste and the edges were beveled. Bars were orientated so that the tensile surface was perpendicular to the hot pressing direction. Twelve bars were tested per processing condition.

Fracture toughness was measured using the controlled flaw indentation technique [20] in 4-point bending on 3.2 X 3.2 X 25.4 mm bars at a crosshead speed of 0.2 mm/minute. The outer span was 19.1 mm and the inner span was 6.4 mm. Tensile surfaces were finished with 1.0 μm diamond paste and the edges were beveled. Three indents were placed on the tensile surface with a Vicker's indenter at a load of 133 newtons. Bars were orientated so that the tensile surface was perpendicular to the hot pressing direction. Twelve bars were tested per processing condition.

Results and Discussion

(1) Whisker Characterization

The SEM and TEM characterization results for the unmodified β-Si_3N_4 whiskers were reported previously [18] and are summarized here. SEM analysis indicated that the whiskers were quite straight, with smooth surfaces. The whiskers were also found to be hexagonal in cross section. TEM stereographic analysis confirmed that the axis of the whiskers coincided with the [0001] crystallographic direction. Further TEM analysis revealed that the single crystal whiskers were of extreme perfection, as few defects, such as dislocations, stacking faults, inclusions, etc., were identified.

The BN modified β-Si_3N_4 whiskers gained approximately one weight percent during the BH_3NH_3 treatment procedure. Calculation of the average deposition thickness, based on the whisker surface area and assuming pure BN, yielded a value of 300Å. As shown in Figure 4, TEM analysis of the modified whiskers revealed a monolithic coating approximately 300Å in thickness. It should also be noted that a portion of the whiskers were cemented together as a result of the surface coating. SEM analysis of the treated whiskers, illustrated in Figure 5, demonstrates that the coating was continuous and smooth. Additionally, no evidence of cracking or spalling was found.

A summary of the XPS results for both the unmodified and BN modified β-Si_3N_4 whisker surfaces is presented in Table IV. For the unmodified whiskers, the survey scan indicated that the surface consisted of silicon (44.01 weight percent), nitrogen (25.47 weight percent), oxygen (14.40 weight percent), yttrium (12.88 weight percent), and carbon (3.24 weight percent). Examination of the detailed spectra revealed that the surface consisted primarily of Si_3N_4 (Si2p peak at 101.54 eV and N1s peak at 397.14 eV), with a minor amount of silicon oxynitride (Si2p peak at 102.94 eV, N1s peak at 398.67 eV, and O1s peak at 531.56 eV). A trace amount of an unidentified compound (O1s peak at 529.93 eV) was also detected. For the BN modified whiskers, the survey scan indicated that the surface consisted of silicon (19.87 weight percent), nitrogen (22.93 weight percent), oxygen (18.63 weight percent), yttrium (2.03 weight percent), boron (25.34 weight percent), and carbon (11.20 weight percent). Examination of the detailed spectra revealed that the surface consisted primarily of Si_3N_4 (Si2p peak at 101.83 eV and N1s peak at 397.70 eV) and BN (B1s peak at 190.65 eV), with a minor amount of silicon oxynitride (O1s peak at 532.39 eV). Surface B_2O_3 (B1s peak at 192.91 eV) and possibly silicon (Si2p peak at 99.80 eV) were identified at trace levels. Two additional unidentified compounds (N1s peak at 396.32 eV and O1s peak at 530.86 eV) were also detected in trace amounts.

(2) Composite Characterization / Mechanical Property Evaluation

Upon hot pressing, the α-Si_3N_4 powder transformed completely to β-Si_3N_4. The strongest peaks for both α-Si_3N_4 and β Si_3N_4 occur between 33° and 37° 2θ. Over this interval, for the powder / whisker mixture prior to hot pressing, the α-Si_3N_4 (102) and (210) peaks due to the powder and the β-Si_3N_4 (101) and (210) peaks due to the whiskers were observed. The α-Si_3N_4 (102) and (210) peaks disappeared after hot pressing, as only the β-Si_3N_4 (101) and (210) peaks were observed.

Table V contains the measured physical and mechanical properties. The percent theoretical density of the polycrystalline Si_3N_4 was 99.1 percent. The addition of 30 volume percent whiskers to polycrystalline Si_3N_4 required higher hot pressing temperatures in order to achieve theoretical densities in the 96 to 98 percent range. The polycrystalline Si_3N_4 exhibited a fracture stress of 649 MPa and a fracture toughness of 3.95 MPa$\sqrt{m}$. Composites containing unmodified β-Si_3N_4 whiskers exhibited the same fracture stress as the unreinforced specimens, but approximately twice the fracture toughness. Composites containing BN modified β-Si_3N_4 whiskers retained approximately 70 percent of the fracture stress of the unreinforced specimens, but still achieved approximately twice the

fracture toughness. The whisker clustering observed during the BN deposition process may lead to flaws in the hot pressed composite, which could lead to a reduction in the fracture stress.

SEM micrographs of the fracture surfaces for the composites produced with the unmodified and BN modified β-Si_3N_4 whiskers are shown in Figures 6 and 7, respectively. Note, that for all the composite specimens produced the matrix grain size was submicron, similar to that of the polycrystalline Si_3N_4. Figure 6 illustrates that there is limited whisker pullout for the composites produced with the unmodified whiskers. In contrast, the composites produced with the BN modified whiskers shown in Figure 7 exhibited an increased amount of whisker pullout, as more whisker troughs were visible on the fracture surfaces. It appeared that the interfacial shear stress was decreased by the presence of the BN on the whisker surfaces.

Summary

The results of this initial investigation into the processing and characterization of β-Si_3N_4 whisker / Si_3N_4 matrix composites are summarized below:

(1) The deposition method described was effective for coating the β-Si_3N_4 whiskers with BN.

(2) The β-Si_3N_4 whisker / Si_3N_4 matrix composites were hot pressed to near theoretical density. The microstructures consisted of submicron β-Si_3N_4 grains surrounding uniformly distributed hexagonal β-Si_3N_4 whiskers.

(3) The fracture stress of the composites produced with the unmodified whiskers was comparable to the polycrystalline β-Si_3N_4 (approximately 630-650 MPa). The fracture stress of the composites produced with the BN modified whiskers decreased 70 percent (approximately 430-450 MPa).

(4) The composites produced with either the unmodified or BN modified whiskers exhibited fracture toughness levels approximately twice that of the polycrystalline β-Si_3N_4 of similar grain size.

(5) Fractography showed strong bonding between the unmodified whiskers and the matrix, while the bonding between the BN modified whiskers and the matrix appeared to decrease to some extent. In all composites produced, the primary toughening mechanism appeared to be

related to whisker bridging and pullout.

Acknowledgments

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Footnotes

* UBE Industries Ltd., Ube City, Japan
** Alfa Chemicals, Morton Thiokol Inc., Danvers, MA
† Fisher Scientific, Springfield, NJ
†† Model 3600, GVA Corp., Vacuum Industries Div., Somerville, MA
# Model 400T, Phillips Instruments, Eindhoven, Netherlands
Model Phi 5400, Perkin-Elmer Corp., Eden Prairie, MN
§ Model R400, Rigaku U. S. A., Danvers, MA
§§ Model S-800, Hitachi, Tokyo, Japan

Table I. Summary of Mechanical Properties of SiC Whisker / Si_3N_4 Matrix Composites.

Whisker Content (Vol. %)	Fracture Stress (MPa)	Fracture Toughness (MPa$\sqrt{m}$)	Test Temperature (°C)	Reference
0	375	4	25	16
20*	550	7	25	16
0	900	6	25	13
10**	625	5.5	25	13
20**	575	5	25	13
0	780	4.7	25	14
30†	970	6.4	25	14
0	575	4.9	1000	14
30†	820	7.5	1000	14
0	480	6.2	1200	14
30†	590	7.7	1200	14
0	660	7.1	25	11
30††	450	10.5	25	11

* Advanced Composite Materials Corp., Greer, SC
** Tokai Carbon, Tokyo, Japan
† Arco Chemical Co., Greer, SC
†† Los Alamos National Lab, Los Alamos, NM

Table II. Chemical and Physical Properties of Raw Materials.

	β-Si_3N_4 Whiskers	α-Si_3N_4 Powder
Phases Present	β-Si_3N_4	α-Si_3N_4
Average Diameter (μm)	1.0	0.2
Average Length (μm)	30	---
Aspect Ratio	30	---
Density (gm/cm^3)	3.19	3.18
Surface Area (m^2/gm)	2.06	17.0
Bulk Chemical Composition (wt. %)		
Silicon	59.1	60.0
Nitrogen	41.3	39.9
Oxygen	0.42	1.41
Yttrium	0.83	<0.003

Table III. Hot pressing conditions.

Hot Pressing Temperature	Hot Pressing Time	Hot Pressing Pressure	Whisker Volume	Whisker Surface
1750°C	2 Hours	30 MPa	0 %	---
1800	2	30	30	Unmodified
1825	2	30	30	Unmodified
1800	2	30	30	BN Modified
1825	2	30	30	BN Modified

Table IV. Summary of XPS results.

Unmodified Whiskers

Orbital	Measured Position*	Compound	Expected Position
C1s	284.6eV	Carbon/ Hydrocarbon	284.6eV [21,22]
Si2p	101.54	Silicon Nitride	101.4-101.7 [22]
Si2p	102.94	Silicon Oxynitride	101.7-103.0 [22]
N1s	397.14	Silicon Nitride	397.4 [22,23]
N1s	398.67	Silicon Oxynitride	397.7-398.4 [23]
O1s	529.93	----------	----------
O1s	531.56	Silicon Oxynitride	532.4-532.7 [23]

* Binding energies are referenced to adventitious carbon at an assumed energy of 284.6eV.

Table IV. (Continued)

BN Modified Whiskers

Orbital	Measured Position*	Compound	Expected Position
C1s	284.6eV	Carbon/ Hydrocarbon	284.6eV [21,22]
Si2p	99.80	Silicon	98.7-99.5 [24]
Si2p	101.83	Silicon Nitride/ Silicon Oxynitride	101.4-101.7 [22] 101.7-103.0 [22]
N1s	396.32	----------	----------
N1s	397.70	Silicon Nitride/ Silicon Oxynitride/ Boron Nitride	397.4 [22,23] 397.7-398.4 [22,23] 398.4-398.5 [24,25]
O1s	530.86	----------	----------
O1s	532.39	Silicon Oxynitride	532.4-532.7 [23]
B1s	190.65	Boron Nitride	190.6-190.9 [24,25]
B1s	192.91	Boron Oxide	193.1 [24]

* Binding energies are referenced to adventitious carbon at an assumed energy of 284.6eV.

Table V. Mechanical properties of polycrystalline Si_3N_4 and β-Si_3N_4 whisker / Si_3N_4 matrix composites.

Hot Pressing Temperature	% Thecretical Density	Fracture Stress	Fracture Toughness
		0 Volume Percent Whisker Content	
1750°C	99.1%	649 (48)* MPa	3.95 (0.75)* MPa$\sqrt{m}$
		30 Volume Percent (Unmodified) Whisker Content	
1800	95.9	659 (77)	8.58 (1.76)
1825	97.3	629 (79)	7.57 (1.24)
		30 Volume Percent (BN Modified) Whisker Content	
1800	95.9	444 (28)	7.99 (0.79)
1825	98.3	428 (38)	9.19 (0.87)

* Standard deviation.

Figure 1. Condensed phases predicted by thermodynamic simulation of Si_3N_4 in the presence of BH_3. (Total pressure = 0.1 MPa)

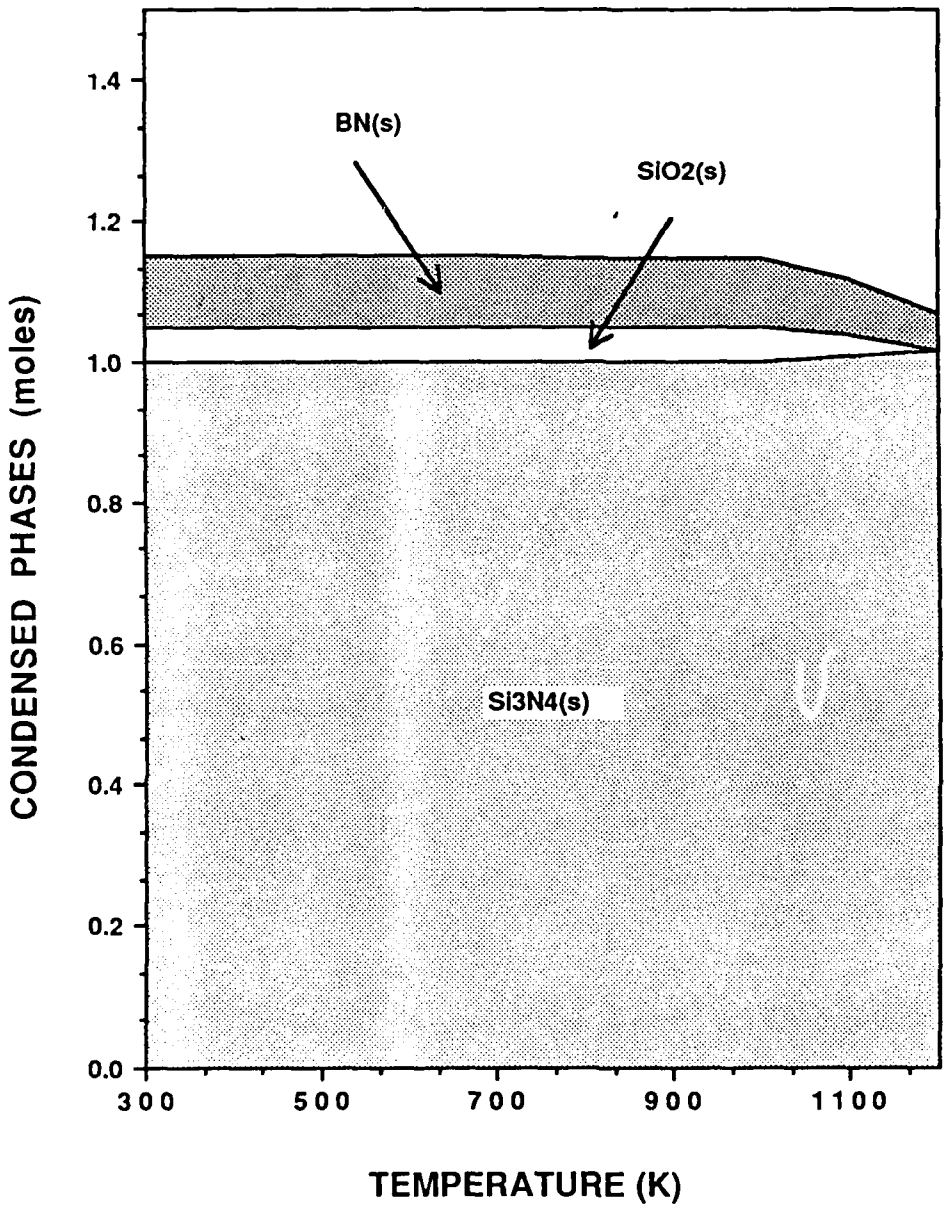

BN(s)
SiO2(s)
Si3N4(s)
CONDENSED PHASES (moles)
TEMPERATURE (K)
0.0
0.2
0.4
0.6
0.8
1.0
1.2
1.4
300
500
700
900
1100

Figure 2. Condensed phases predicted by thermodynamic simulation of Si_3N_4 in the presence of MgO. (Total pressure = 30.0 MPa)

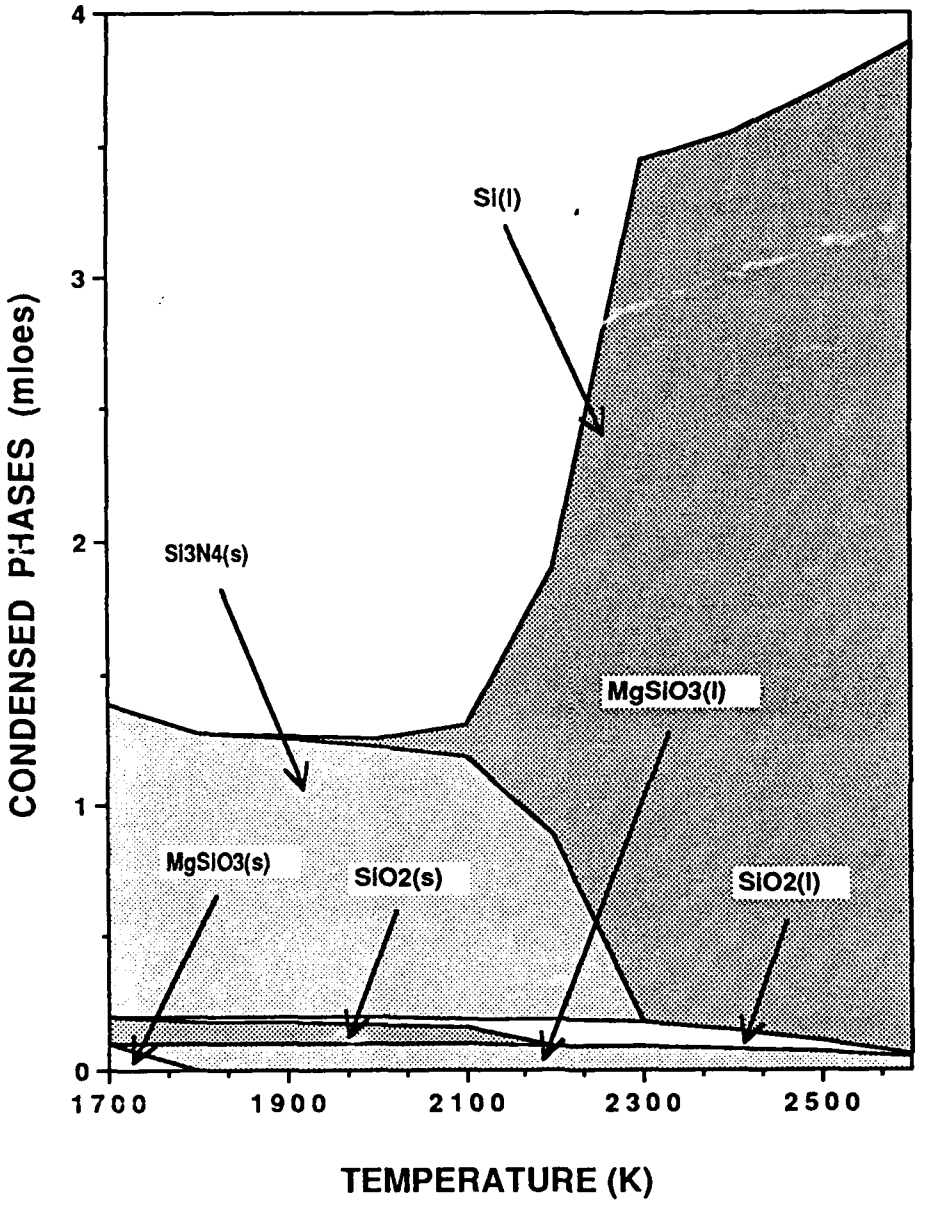
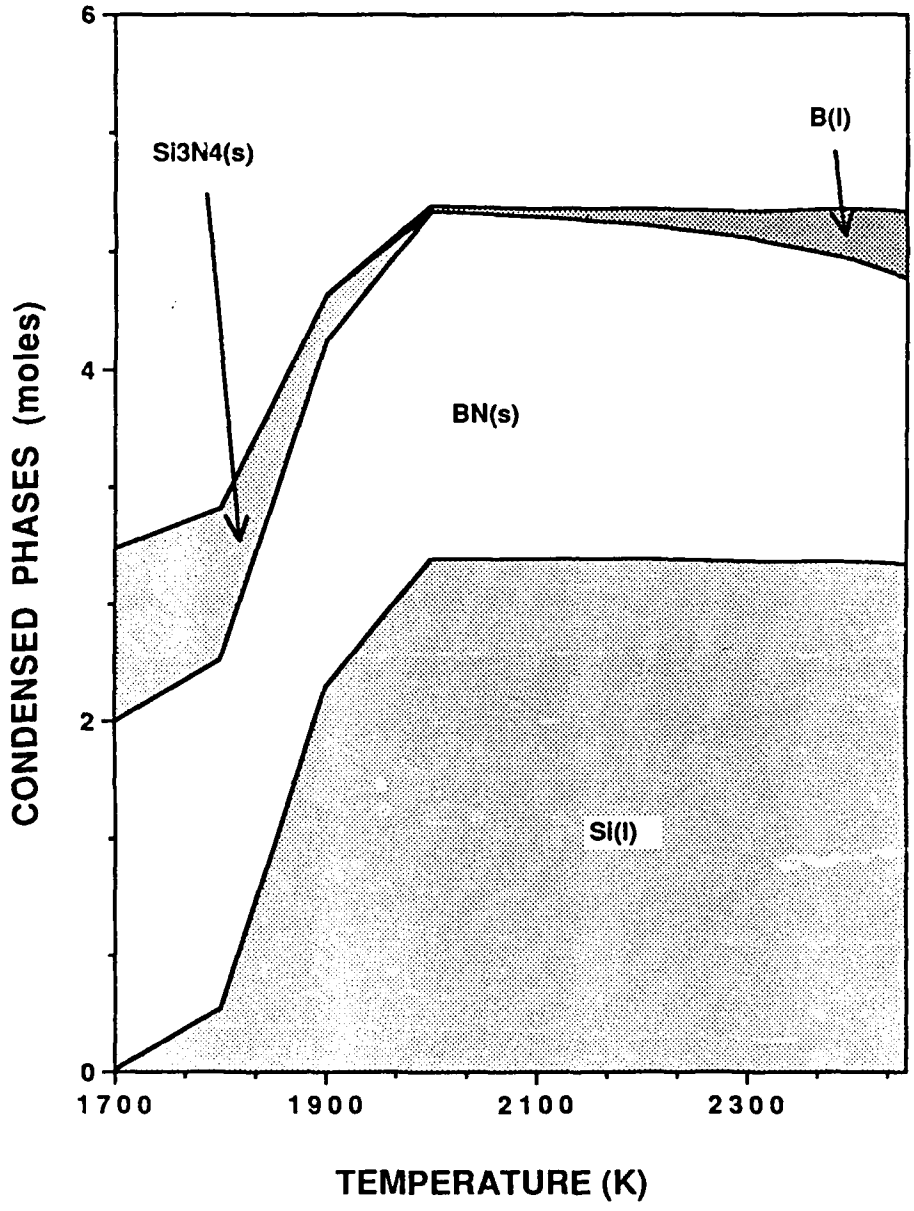

CONDENSED PHASES (moles)
4
3
2
1
0
1700
1900
2100
2300
2500
TEMPERATURE (K)
Si(l)
Si3N4(s)
MgSiO3(l)
MgSiO3(s)
SiO2(s)
SiO2(l)

Figure 3. Condensed phases predicted by thermodynamic simulation of Si_3N_4 in the presence of BN. (Total pressure = 30.0 MPa)


6
4
2
0
Si3N4(s)
B(l)
BN(s)
Si(l)
1700
1900
2100
2300
CONDENSED PHASES (moles)
TEMPERATURE (K)

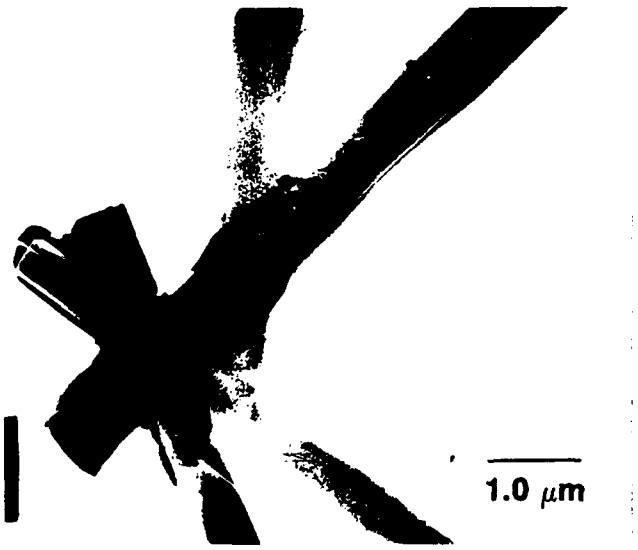


Figure 4. TEM micrograph of BN modified β-Si_3N_4 whiskers.

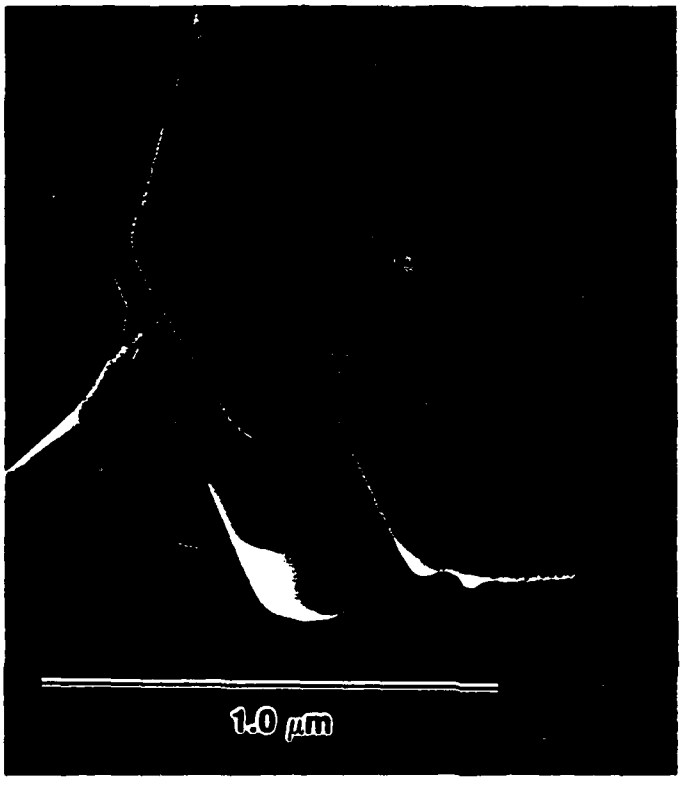


Figure 5. SEM micrograph of BN modified β-Si_3N_4 whiskers.

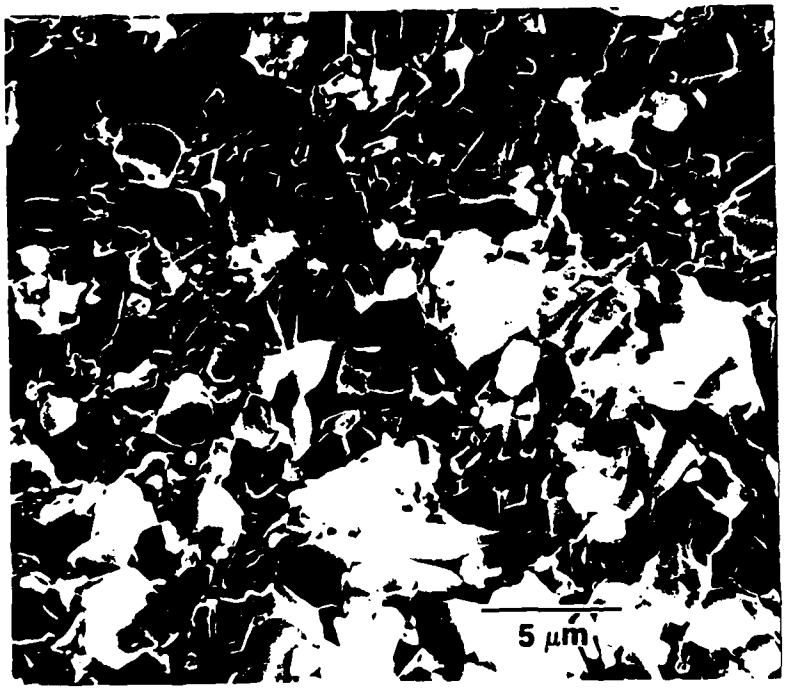


Figure 6. SEM micrograph of fracture surface of composite produced with unmodified β-Si_3N_4 whiskers. (Hot pressing temperature = 1852°C)

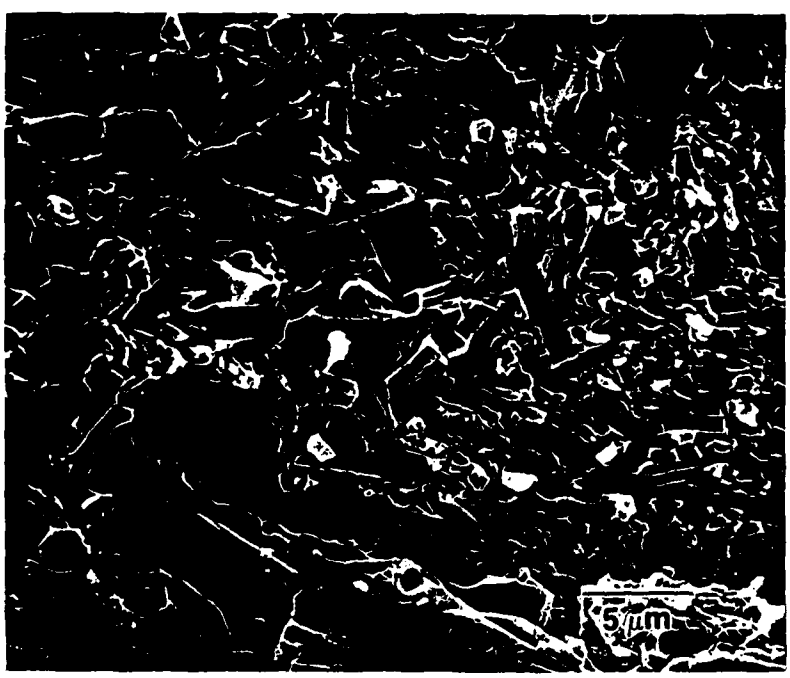


Figure 7. SEM micrograph of fracture surface of composite produced with BN modified β-Si_3N_4 whiskers. (Hot pressing temperature = 1852°C)